भारतीय मानक

Indian Standard

IS 1448 (Part 138) : 2023

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 138 साबुन अंश का निर्धारण

(दूसरा पुनरीक्षण)

Petroleum and its Products — Methods of Test

Part 138 Determination of Soaap Content

(Second Revision)

ICS 75.100

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September 2023 **Price Group 4**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Part 138) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Soap is the primary thickener for most of the greases and properties of grease are largely determined by the type of thickener.

This standard was originally published in 1992 and was based on ASTM D128-89. Subsequently, first revision was done in 2018. In the first revision, calculation of soap with single grease based and soap with mixed grease based were included and necessary assistance had been derived from ASTM D128 : 1998 and reapproved in 2014.

The second revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major change has been incorporated:

a) Reference and procedure clauses have been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

PART 138 DETERMINATION OF SOAP CONTENT

(Second Revision)

1 SCOPE

This standard (Part 138) prescribes the method of test for the determination of soap content in lubricating greases of the conventional type, which consists essentially of petroleum oil and soap and also filler, asphalt, residuum and tars.

2 REFERENCES

The standards given below contain provisions which, through reference in text constitute provisions of this standard. At the time of publication, the edition indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 170 : 2020	Acetone — Specification (*fifth revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 336 : 2021	Ether — Specification (*third revision*)
IS 3470 : 2017	Hexane, food grade — Specification (*second revision*)

3 SAMPLE

3.1 The sample size for the soap determination shall be from 8 g to 30 g, depending on the consistency of the grease, which is chiefly determined by the percentage of soap content. A 10 g to 20 g sample is usually convenient amount for NLGI (National Lubricating Grease Institute, USA) No. 3 consistency greases, while softer greases require a large sample. The original sample shall be stirred or mixed until uniform.

3.2 Samples need not be weighed more closely than to 0.1 g.

4 REAGENTS

4.1 *n*-Hexane — Passing the blank test prescribed below (*see* IS 3470)

Boil 125 ml of n-hexane with 10 ml of 0.5 N alcoholic KOH solution and 50 ml of neutral 50 percent alcohol for 1.5 h on a hot plate. Use a glass tube about 7 mm inside diameter and 750 mm in length as a reflux condenser. After cooling titrate the solution with 0.5 N HCl using phenolphthalein indicator. Not less than 9.8 ml of 0.5 N HCl shall be required for neutralization.

4.2 Dilute Hydrochloric Acid (HCl)

A solution containing 10 percent by weight of the concentrated HCl in water with a permissible variation of 0.5 percent.

4.3 Standard Alcoholic Potassium Hydroxide (KOH) Solution — 0.5 N

4.4 Rectified Spirit — Neutralized to phenolphthalein (*see* IS 323)

4.5 Concentrated Hydrochloric Acid (HCl) — Relative density 1.19 (*see* IS 265)

4.6 Phenolphthalein Indicator Solution

The phenolphthalein solution shall be prepared by dissolving 1.0 g of phenolphthalein in 50 ml of rectified spirit adding 5 ml of water and neutralizing with NaOH or KOH solution.

CAUTION — Avoid skin contact or ingestion.

4.7 Diethyl Ether — (*see* IS 336)

4.8 Acetone — (*see* IS 170)

5 PROCEDURE

Analyze greases containing residuum asphaltic oils and asphalt, tars, etc using Method B, as these ingredients usually cause stubborn emulsions if a vigorous agitation process is applied at the start. Analyze all other greases using Method A.

5.1 Method A

5.1.1 Weigh the grease sample (*see* **3.1**) into either 250 ml or 500 ml Erlenmeyer flask, spreading the sample over the lower inside surfaces of the flask. Add 20 ml of *n*-hexane followed by 50 ml of HCl (10 percent) and several boiling chips. Digest the sample by boiling under a reflux condenser until it is completely decomposed. Formation of a clear oil layer is evident of complete decomposition. A digestion time of 3 h is generally sufficient. However, more vigorous boiling or use of a stronger HCl solution or additional *n*-hexane may be required for greases difficult to decompose. Allow the content of the flask to cool to room temperature.

5.1.2 Remove the condenser. If insolubles appear, filter the content of the flask, while they are warm, through a Gooch crucible provided with a mat. The residue on the Gooch crucible is washed thoroughly with warm water and hexane, and dried to account for insolubles present in the greases.

NOTE — If insolubles are absent, the filtration of the decomposed grease is avoided.

5.1.3 Transfer the content of the flask into a separating funnel (500 ml) using *n*-hexane and distilled water as washing liquids.

5.1.4 Allow the solution in the separating funnel to clear (*see* Note) and then draw off the aqueous layer 'A' into another 250 ml separating funnel.

NOTE — In case two layers are observed in the *n*-hexane portion, the layer can be broken by addition of 10 ml rectified spirit.

Wash the *n*-hexane layer 'B' three times with 25 ml distilled water to remove hydrochloric acid. Add the aqueous washings to layer 'A' in 250 ml separating funnel. Extract the aqueous solution 'A' thrice with 25 ml of *n*-hexane extracts 'C' and wash 'C' with 15 ml of distilled water and add the washed *n*-hexane extracts 'C' to 'B' in the 250 ml separating funnel.

5.1.5 Titrate the *n*-hexane extract 'B' if comparatively light coloured, with standard 0.5 N alcoholic potassium hydroxide using phenolphthalein indicator and determine approximately the free fatty acids and fatty acids from soaps, taking 200 as the average neutralization value of the fatty acid (that is, 1.0 g of fatty acid requires 200 mg of potassium hydroxide for neutralization). If, however, the n-hexane extract 'B' is dark, add a few drops of phenolphthalein and sufficient quantity of standard potassium hydroxide solution to make the alcoholic layer distinctly alkaline after vigorous shaking. If the n-hexane extract 'B' has been titrated, add a slight excess of alkali before separating (careful addition of rectified spirit at this point may aid in securing rapid and sharp separation).

5.1.6 Allow the solution to separate sharply and draw off the lower alcoholic layer 'D' into another separator. Wash the upper *n*-hexane layer 'E' three times with 30 ml, 25 ml and 20 ml of neutral 50 percent alcohol respectively and add these washes to 'D'. Wash solution 'D' with 25 ml of *n*-hexane, after which draw off 'D' into a beaker and add the *n*-hexane to 'E'. Evaporate solution 'D' to a small volume to remove alcohol, wash the residue of potassium soap into a separator with hot water, acidify with HCl and shake out twice with 50 ml and 25 ml of ethyl ether 'F' respectively. Run solution 'F' into another separator and wash twice with 20 ml portions of water which may then be discarded.

5.1.7 Transfer 'F' into a weighed beaker and evaporate to dryness on a steam bath, blowing with air to remove all traces of ethyl ether. Heat the residue consisting of free fatty acids and fatty acids from soap for a short time on a steam bath, adding and evaporating 5 ml portions of acetone until a constant weight is obtained and the last traces of water are removed. Determine the exact neutralization value on as large a sample of these fatty acids as possible. The fatty acids may be identified to some extent by special tests such as odour, crystal form, melting point, iodine number, colour reactions, etc.

5.1.8 If the grease sample is appreciably oxidized, the fatty acids obtained by the ethyl ether extraction are likely to be dark in colour and hard to identify. For further study and identification, the neutralized acids be extracted again (qualitatively) with a mixture of one part *n*-hexane and 3 parts HCl, thus eliminating the small amount of more darkly coloured matter.

5.1.9 From the total quantity of fatty acid found, deduct the free fatty acid if any and calculate the remainder as percentage of soap as given in **6**.

5.2 Method B

Weigh the sample (*see* **5.1.1**) in a 76 mm porcelain dish and add 10 g of granulated KHSO4, 10 g clean dry ignited sand and 5 ml of distilled water. Heat the dish and contents on a steam bath while stirring frequently, until all water is driven off, 2 h is usually sufficient for this. After cooling and breaking up the lumps with a small pestle, transfer the mixture quantitatively to an extraction thimble, which has been previously placed in a Soxhlet apparatus using a little *n*-hexane to wash the last traces into the thimble. Extract the thimble with *n*-hexane, concentrate the extract if necessary and titrate the free fatty acid and fatty acid from the soap with 0.5 N alcoholic KOH solution as in **5.1.4**. Proceed further as in **5.1.5** to **5.1.9**.

6 CALCULATION

6.1 Calculate and report the neutralization number, mean molecular weight of fatty acids, and percentage of soap as specified under **6.2**, **6.3** and **6.4** respectively.

6.2 Neutralization number of fatty acids:

$$A = \frac{56.1 \times B \times N}{M}$$

where

B = volume, in ml, of standard alcoholic potassium hydroxide solution required for titration;
N = normality of the standard potassium hydroxide solution; and
M = mass, in g, of fatty acid that were titrated.

6.3 Mean molecular weight of fatty acids:

$$= \frac{56.1}{A} \times 1\,000$$

where

A = neutralization number of fatty acids.

6.4 Soap, percent by mass:

$$= \frac{D \times E}{C \times F}$$

where

D = mean molecular weight of the soap;
E = percentage of fatty acids from soap;
C = mean molecular weight of fatty acids from soap; and
F = number of moles of fatty acid per mole of soap.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray **(*Chairperson*)**
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